

# Supporting information for:

## Thermal Route for the Synthesis of Maghemite/Hematite Core / Shell Nanowires

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## Protocol for the biological study

In the present work, U373 glioblastoma cell line sed comes from the American Type Culture Collections (Manassas, VA, USA). The cell line was grown in Dulbecco's Modified Eagle's Medium (DMEM) supplemented with 10% of fetal bovine serum (FBS), 2mM L-glutamine, 1 $\mu$ g/ml fungizone, 100 $\mu$ g/ml streptomycin and 100 U/ml of penicillin. These media were supplied from GIBCO. Cell line was maintained at 37 °C in a humidified atmosphere of 95% air and 5% CO<sub>2</sub>.

For the internalization of iron oxide NWs, they were sterilized before cell incubation using UV light for 1 h. Then, the solution was dispersed by sonication for 5 min and NWs were mixed with DMEM 10% FBS. For direct incubation internalization (DI) cells were plated in a 24 well plate at  $2.5 \times 10^4$  cells per well in 500  $\mu$ l of DMEM containing 10% FBS. After 24 h, the growth medium was removed and cells were mixed with iron oxide NWs dispersed in different concentrations, from 1000 to 30000 NWs by cell. For the centrifugation-mediated internalization (CMI) method,  $5 \times 10^4$  glioblastoma cells were plated in tubes of 15 ml and centrifuged at 1000 rpm for 5 min. The supernatant was removed and 500  $\mu$ l of iron oxide NWs solution at different concentration were mixed and centrifuged at 1500 rpm for 5 min. Then, the medium was exchanged with fresh medium and cells were plated in a 24 well plate and maintained at 37°C for 24 h. After carrying out the DI and CMI methods, cells were washed two times with PBS (AMRESCO, Ohio, USA) and fixed with 4% paraformaldehyde solution for 30 min. After that, cells were washed twice with PBS, and were incubated with prussian blue solution. This solution contains a 1:1 mixture of 4% Potassium Ferrocyanide (Sigma-Aldrich) and 4% hydrochloric acid (Sigma-Aldrich). Samples remained in the prussian blue solution for 15 min and washed three times with distilled water. On the other hand, the cytoplasm was marked with neutral red 0.5% (Panreac Quimica S.L.U) for 2 min and washed with distilled water twice. Once samples were dried, the cover slip was mounted using the mounting medium DePex (SERVA Electrophoresis GmbH).

## SEM images of different iron oxides annealed under different conditions

SEM images of iron oxides NWs obtained under different annealing conditions are shown in Figure S1. Modifying annealing times and temperatures of Fe NWs, the density of the final iron oxide NWs remains unchanged but some morphological features vary: for example, the diameter is larger as we increase the annealing temperature and annealing times.

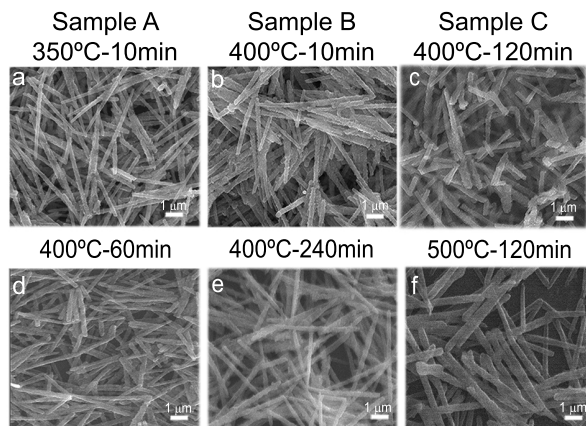


Figure S1: SEM images of NWs prepared under different annealing times and temperatures. a) Sample A, 350°C for 10min, b) Sample B, 400°C for 10 min, c) Sample C, 400°C for 120 min, d) 400°C for 60 min, e) 400°C for 240 min and f) 500°C for 120 min.

## XRD patterns of Samples A, B and C

X-Ray Diffraction (XRD) measurements were carried out using a Philips X'Pert PRO system provided with a Cu target ( $\lambda_{\text{Cu}}=1.54 \text{ \AA}$ ) and a four-circle goniometer PW3050/60. Samples were measured in grazing incidence mode (GIXRD) and the detector scanned in the  $2\theta$  range from  $20^\circ$  to  $90^\circ$ . The X-ray incident grazing angle for measurements was fixed at  $0.4^\circ$ .

The diffraction maxima have been indexed as 104, 110, 113, 024, 116, 214 and 300 reflections corresponding to the rhombohedral phase of hematite (ICDD -01-071-5088) and to 311, 422, 511 and 440 reflections corresponding to the maghemite cubic phase (ICDD

00-039-1346). The maximum marked with an \* in the pattern corresponds to the Si (100) substrate reflection.

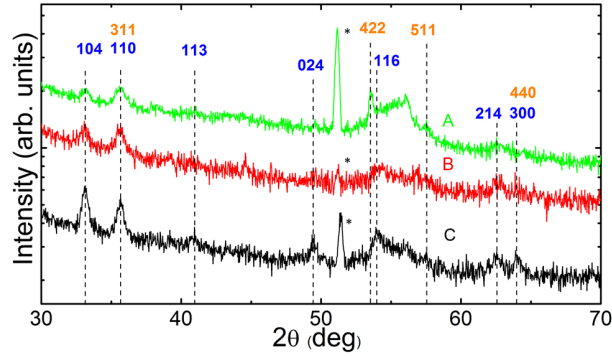


Figure S2: XRD patterns collected from sample A (green), sample B (red) and sample C (black). Hematite diffraction maxima are marked in blue while maghemite maxima are marked in orange. The (100) diffraction maximum corresponding to the Si substrate is marked with \* in the figure.

## Analysis of Raman spectroscopy

We have applied Fourier-Self Deconvolution (FSD) method in order to narrow the Raman bands and differentiate between hematite and maghemite vibrational modes in a clearer way. For the FSD analysis we have taken in consideration two factors: the width of the natural lineshape ( $\sigma$ ), and the apodization function for the improvement of the spectral resolution, which is based on the width ( $\Delta$ ). The most critical parameter is the  $\sigma$  of the Lorentzian lineshape: when the bandwidth is overestimated, a spectrum with larger oscillations and negative side-lobes is obtained; meanwhile the underestimation of this value results in a FSD spectrum with a much lower resolution. In order to reach the best compromise between spectral resolution and the deconvolution of the Raman modes we changed the parameters  $\sigma$  and  $\Delta$  as follows: Sample A,  $\sigma=47 \text{ cm}^{-1}$  and  $\Delta=38 \text{ cm}$ ; Sample B,  $\sigma=50 \text{ cm}^{-1}$  and  $\Delta=38 \text{ cm}$ ; Sample C,  $\sigma=70 \text{ cm}^{-1}$  and  $\Delta=38 \text{ cm}$  and finally for hematite NWs,  $\sigma=24 \text{ cm}^{-1}$  and  $\Delta=50 \text{ cm}$ .

We show the original Raman spectrum (Figure S3.a) of each sample together with

the FSD spectra (Figure S3.b) where the Raman modes as  $E_g(5)$  of hematite and  $A_{1g}$  of maghemite oxide, used for the calculation of the maghemite/hematite ratio in the NWs, are marked.

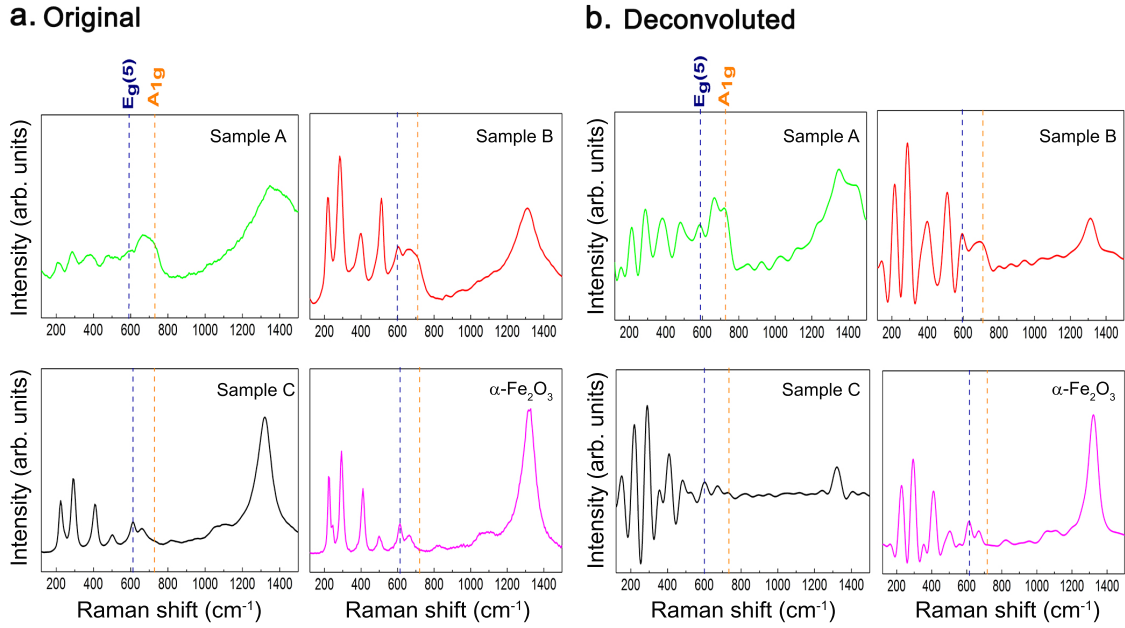


Figure S3: a) Original Raman spectra and b) Fourier-Self Deconvolution of the Raman spectra for samples A, B, C and the hematite NWs in the spectral range from  $100\text{cm}^{-1}$  to  $1500\text{ cm}^{-1}$ . Raman modes as  $E_g(5)$  for hematite and  $A_{1g}$  for maghemite are marked with a blue and orange line respectively.